

PATENT COOPERATION TREATY

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NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
 United States Patent and Trademark
 Office
 Box PCT
 Washington, D.C.20231
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

| | |
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| Date of mailing (day/month/year) 09 August 2000 (09.08.00) | |
| International application No. PCT/GB99/04375 | Applicant's or agent's file reference JDM/P. 401111 WO |
| International filing date (day/month/year) 23 December 1999 (23.12.99) | Priority date (day/month/year) 24 December 1998 (24.12.98) |
| Applicant CASPER, James, William | |

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

27 May 2000 (27.05.00)

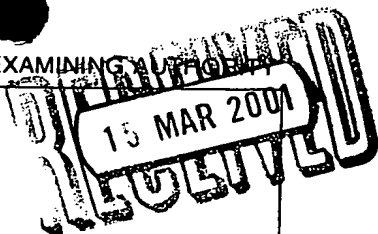
☐ in a notice effecting later election filed with the International Bureau on:2. The election ☒ was☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

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| The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35 | Authorized officer S. Mafla Telephone No.: (41-22) 338.83.38 |
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PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY



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NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT Rule 71.1)

To:

W.P. THOMPSON & CO.
Coopers Building
Church Street
Liverpool L1 3AB
GRANDE BRETAGNE

Date of mailing
(day/month/year) 13.03.2001

Applicant's or agent's file reference
JDM/P. 401111 WO

IMPORTANT NOTIFICATION

International application No.
PCT/GB99/04375

International filing date (day/month/year)
23/12/1999

Priority date (day/month/year)
24/12/1998

Applicant
PPG INDUSTRIES OHIO, INC. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Connolly, M

Tel. +49 89 2399-8021





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| Applicant's or agent's file reference JDM/P. 401111 WO | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/GB99/04375 | International filing date (day/month/year) 23/12/1999 | Priority date (day/month/year) 24/12/1998 |
| International Patent Classification (IPC) or national classification and IPC C09D17/00 | | |
| Applicant PPG INDUSTRIES OHIO, INC. et al. | | |

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 4 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

| | |
|---|--|
| Date of submission of the demand 27/05/2000 | Date of completion of this report 13.03.2001 |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer Van de Panne, V Telephone No. +49 89 2399 8405  |

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/04375

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).):*

Description, pages:

1-14 as originally filed

Claims, No.:

1-24 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/04375

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | | |
|-------------------------------|------|--------|-------------|
| Novelty (N) | Yes: | Claims | 1-18, 20-23 |
| | No: | Claims | 19, 24 |
| Inventive step (IS) | Yes: | Claims | 1-18, 20-23 |
| | No: | Claims | |
| Industrial applicability (IA) | Yes: | Claims | 1-24 |
| | No: | Claims | |

2. Citations and explanations see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/04375

I The documents cited in the search report do not disclose or suggest any compositions in which an aqueous pigment dispersion is present as the water phase in a W/O emulsion wherein the continuous phase is a solution of a polymer having functional groups and hydrophilic groups in an organic solvent. Nor do they disclose the addition of a crosslinker to such a composition or the preparation of such a composition. In spite of the X-classification of all documents cited in the search report, the subject-matter of the independent claims 1, 20 and 23 and of the dependent claims 2-18, 21 or 22 is novel and involves an inventive step.

II US-A 3 959 554 ("D1") discloses aqueous dispersions for use in coating, comprising a pigment, an acrylic polymer having both hydroxylic groups and carboxylic groups which also acts as the pigment dispersant in the preparation of the mill base and a crosslinker. In view of the manner of preparation of the dispersion, it will contain a certain amount of organic solvent, just as is the case in the dispersions of claim 19. See D1, column 1 lines 38-69, column 3 lines 11-30, column 4 lines 8-15 and working example 1. Although the present invention arrives at the dispersion in a different manner, the same components are present in the dispersion as in the dispersion of D1, and this disclosure takes away the novelty of the present claims 19 and 24.

These claims also lack novelty in view of the disclosure of DE-A 4 226 270 ("D2"), which also arrives by different means at compositions indistinguishable from those of claim 19 and at the coated object of claim 24. This document discloses aqueous coating compositions comprising a dispersed polymer which possesses hydroxylic groups and carboxylic groups, a diisocyanate as the crosslinker and a pigment with a pigment dispersant. As the solvent used in the preparation of the polymer and the solvent which may be used to liquefy the polyisocyanate are not removed, the dispersion may contain a non-negligible amount of organic solvent. See D2, claims 1, 2, 4, 8 and 9, page 2 line 65 to page 3 line 15, page 6 lines 40-44 and 57-58, page 9 lines 11-15 and lines 37-39 and working example 10.

The remaining documents are less relevant and do not need to be discussed.

PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

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| Applicant's or agent's file reference JDM/P. 401111 WO | FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below. | |
| International application No. PCT/GB 99/ 04375 | International filing date (day/month/year) 23/12/1999 | (Earliest) Priority Date (day/month/year) 24/12/1998 |
| Applicant PPG INDUSTRIES OHIO, INC. et al. | | |

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.
☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the title,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

- ☐ as suggested by the applicant.
- ☐ because the applicant failed to suggest a figure.
- ☐ because this figure better characterizes the invention.
- ☐ None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No

P 99/04375

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C09D17/00 C08G18/62

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C09D C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|------------|---|---------------------------|
| X | EP 0 358 979 A (BAYER) 21 March 1990 (1990-03-21) page 2, line 37 -page 6, line 21; claims 1-6; examples --- | 1-6, 8-10, 16-24 |
| X | WO 96 30425 A (PPG INDUSTRIES) 3 October 1996 (1996-10-03) page 2, line 28 -page 7, line 30; claims 1,7,17; examples --- | 1,5, 16-24 |
| X | DE 42 26 270 A (HERBERTS) 10 February 1994 (1994-02-10) page 2, line 32 -page 9, line 65; claims 1-11; examples --- -/-- | 1-6, 8-10,13, 16-24 |



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

23 March 2000

Date of mailing of the international search report

31/03/2000

Name and mailing address of the ISA

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Authorized officer

Bourgonje, A

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/04375

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|------------|---|------------------------|
| X | DE 35 46 594 A (SYNTHOPOL CHEMIE) 10 December 1987 (1987-12-10) page 3, line 18 -page 6, line 18 --- | 1-6, 8-10, 16-24 |
| X | FR 2 301 575 A (BASF) 17 September 1976 (1976-09-17) page 1, line 30 -page 8, line 14; claims 1,2; examples --- | 1 |
| X | US 3 959 554 A (HICK) 25 May 1976 (1976-05-25) column 1, line 38 -column 4, line 48; examples ----- | 1 |

INTERNATIONAL SEARCH REPORT

Info on patent family members

International Application No

P GB 99/04375

| Patent document cited in search report | | Publication date | Patent family member(s) | Publication date |
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| (51) International Patent Classification ⁷ : C09D 17/00, C08G 18/62 | A1 | (11) International Publication Number: WO 00/39228 (43) International Publication Date: 6 July 2000 (06.07.00) |
| (21) International Application Number: PCT/GB99/04375 (22) International Filing Date: 23 December 1999 (23.12.99) (30) Priority Data: 9828443.3 24 December 1998 (24.12.98) GB (71) Applicant (for all designated States except US): PPG INDUSTRIES OHIO, INC. [US/US]; 3800 West 143rd Street, Cleveland, OH 44111 (US). (72) Inventor; and (75) Inventor/Applicant (for US only): CASPER, James, William [GB/GB]; 24 Parrs Road, Stokenchurch, High Wycombe, Buckinghamshire HP14 3QF (GB). (74) Agent: W.P. THOMPSON & CO.; Coopers Building, Church Street, Liverpool L1 3AB (GB). | | (81) Designated States: AU, BR, CA, JP, MX, US, ZA, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i> |
| (54) Title: COATING COMPOSITION (57) Abstract A pigmented solventborne paint pack which can be made into a waterborne coating composition which comprises; i) a waterborne pigment dispersion, comprising pigment in dispersion in water in the presence of a pigment dispersant, the waterborne pigment dispersion itself being in dispersion in, ii) a solution in an organic solvent of polymer having functional groups and hydrophilic groups. Also, a solventborne activated paint pack which comprises the solventborne paint pack and a crosslinker which is dissolved in the organic solvent and a waterborne coating composition which comprises a dispersion in an aqueous medium of the solventborne activated paint pack. | | |

FOR THE PURPOSES OF INFORMATION ONLY

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-1-

COATING COMPOSITION

This invention relates to a coating composition, certain intermediate compositions in its preparation, a process for preparing the composition and to the use of the composition in a coating process.

One significant market for paints is in the repainting of motor vehicles after crash damage repair or refurbishment. This is often referred to as Vehicle Refinish. One of the most common types of vehicle refinish paints currently on the market comprises a hydroxyl functional polymer and a polyisocyanate crosslinker in an organic solvent. Because the polymer and the crosslinker react together from the time they are mixed, they are generally supplied to the end user as two separate components which are mixed together shortly before use. For this reason, they are often called "two-pack" paints.

In practice, the component containing the polymer also contains any components such as pigments, fillers, extenders or other additives which may be required to give the paint its final colour or to modify its properties. Pigments or extenders are particulate materials which are held in stable dispersion in the solventborne composition by a pigment dispersant. In making the compositions, the pigments, fillers and extenders are usually firstly made into a pigment millbase. A pigment millbase is made by mixing the pigments, fillers and extenders and the pigment dispersant, usually with a little solvent, in a high shear mixer such as a ball mill so as to thoroughly separate the pigment particles and coat them with dispersant. The resulting millbase is then mixed with polymer and solvent to form the composition known as the "paint pack" as sold to the end user. The user then mixes this paint pack with a solution of the crosslinker in organic solvent, referred to as the "hardener", just prior to use.

There is environmental and legislative pressure to reduce the emission of organic solvents from coating compositions when they are applied and dried to form a paint film. One way to achieve this is to use waterborne compositions.

Producing a waterborne equivalent of the vehicle refinish two pack paint described above is not simply a question of replacing all of the solvent with water. Each component must be modified in order to make it water soluble or dispersible. In particular, the modification would necessarily involve

-2-

the introduction of hydrophilic groups into the crosslinker which must be made water dispersible. The introduction of hydrophilic groups into the crosslinker has been found to make the final paint film more sensitive to water, rendering this route unattractive in producing high performance refinish paints with good water resistance.

In an alternative (see EP-358979) the polymer is produced in an organic solvent. On completion of the polymerisation reaction the organic solvent is removed and the polymer is made into an aqueous solution or dispersion. Pigment may then be added to the aqueous polymer solution or dispersion (if required) before addition of the isocyanate crosslinkers.

It is however desired to obtain coatings having improved water resistance compared to the water resistance of coatings produced in accordance with EP-358979. This is achieved in accordance with the present invention by the use of a solution in an organic solvent of polymer having functional group and hydrophilic groups having dispersed therein a waterborne pigment dispersion.

One proposed alternative method of producing a waterborne two pack composition uses a solventborne paint pack. The polymer and the crosslinker are both chosen to be soluble in an organic solvent and the pigment millbase is made using a pigment dispersant compatible with the solvent. The polymer is dissolved and the pigment is dispersed in the organic solvent to form the solventborne paint pack. The end user dissolves the crosslinker in the solventborne paint pack to form an activated paint pack and then disperses this in water to form the waterborne coating composition ready for use.

It has been found that this route can provide waterborne two pack compositions with equivalent performance to solventborne compositions. The problem with this route is that although the majority of the carrier fluid can be water, there is still a relatively high level of solvent present from the solventborne paint pack. Levels of solvent in essentially waterborne compositions are often expressed as volatile organic content (voc) in grams per

litre of composition excluding water (g/l). For primer composition, these types of composition still have a voc in the region of 350g/l. This is less than conventional solventborne primer compositions but existing and forthcoming regulations are aiming at 250g/l, and a still lower level is clearly desirable. For single layer topcoat compositions 5 these types of composition still have a voc in the region of 420g/l, which again is less than conventional solventborne topcoat compositions but forthcoming regulations are aiming at 340g/l, and a still lower level is clearly desirable.

We have now discovered that it is possible to reduce the voc of these types of composition by the use of a waterborne pigment millbase. This waterborne millbase is dispersed in a 10 solution of the polymer in an organic solvent so as to form the paint pack. The polymer has hydrophilic groups so that it acts to stabilise the water-in-oil emulsion of the millbase in the polymer solution. It also has functional groups which will react with the crosslinker. A crosslinker is used which is not hydrophilically modified and which is soluble in the organic solvent. Just prior to use, the crosslinker is added to this dispersion and dissolved 15 in the solvent and then this mixture is finally dispersed in water. This oil-in-water dispersion is also stabilised by the polymer. This method allows crosslinker without hydrophilic groups to be used, while at the same time allowing compositions to be made having a volatile organic content significantly lower than those made by conventional routes.

20 According to the present invention there is provided a pigmented solventborne paint pack which can be made into a waterborne coating composition which comprises;

- i) a waterborne pigment dispersion, comprising pigment in dispersion in water in the presence of a pigment dispersant, the waterborne pigment dispersion itself being in dispersion in
- 25 ii) a solution in an organic solvent of polymer having functional groups and hydrophilic groups.

The present invention also provides a solventborne activated paint pack which comprises the solventborne paint pack and a crosslinker which is dissolved in the organic solvent.

The present invention also provides a waterborne coating composition which comprises a dispersion in an aqueous medium of the solventborne activated paint pack.

According to the present invention there is also provided a process for producing a solventborne paint pack which can be made into a pigmented waterborne coating composition comprising a polymer having functional groups and hydrophilic groups and a crosslinker for the polymer, comprising the steps of;

- i) forming a solution of the polymer in an organic solvent
- ii) dispersing a waterborne pigment dispersion in the polymer solution.

The present invention also provides a process for producing a solventborne activated paint pack, which can be made into an aqueous coating composition, comprising the further step of adding to the solventborne paint pack a crosslinker which is soluble in the organic solvent and forming a solution of the crosslinker in the solvent.

The present invention also provides a process for producing an aqueous coating composition which comprises the further step of emulsifying the solventborne activated paint pack in an aqueous medium.

Organic solvents include any non-aqueous solvents which can be used to dissolve the polymer and which have little or no solubility in water. It can be an aliphatic or aromatic hydrocarbon such as Solvesso 100TM, toluene or xylene, an alcohol such as butanol or isopropanol, an ester such as butyl acetate or ethyl acetate, a ketone such as acetone, methyl isobutyl ketone or methyl ethyl ketone, an ether, an ether-alcohol or an ether-ester or a mixture of any of these

The polymer can be any polymer having functional groups and hydrophilic groups and which can act to stabilise the dispersion of aqueous millbase in the polymer solution and which can stabilise the emulsion of activated paint pack in the aqueous phase.

Functional groups are groups which can react with a crosslinker so as to crosslink the polymer in the final coating film. For example, the functional groups can be amine groups, hydroxyl groups, acetoacetate groups, silane groups, carboxylic acid groups or epoxy groups, preferably hydroxyl groups or epoxy groups and most preferably hydroxyl groups.

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Examples of suitable hydrophilic groups are carboxylic acid groups and amine groups. When the hydrophilic groups are carboxylic acid groups, the polymer preferably has an acid value of 20 to 250. When the hydrophilic groups are amine groups, the polymer preferably has an amine value of 20 to 250. Preferably acid or amine groups on the polymer are at least partially neutralised and more preferably fully neutralised. For example, acid groups can be neutralised using a suitable base such as ammonia or an amine such as dimethylethanolamine, and amine groups can be neutralised using an acid such as lactic acid or acetic acid. When the hydrophilic groups are amine groups or carboxylic acid groups, they can also act as functional groups.

The polymer can be a vinyl addition polymer, a polyester, a polyurethane, a mixed polyester-polyurethane or an epoxy polymer, preferably a vinyl addition polymer, a polyester, a polyurethane or a mixed polyester-polyurethane and most preferably a vinyl addition polymer.

Preferred polymers have a number average molecular weight as measured by gel permeation chromatography of 700 to 10,000, more preferably 1,000 to 4,000. Preferably, when the polymer has hydroxyl groups, it has a hydroxyl value of 5 to 500, more preferably 50 to 250.

Preferred polymers have an acid value (AV) of up to 50. Acid value is the mass of potassium hydroxide in milligrams required to neutralize the acid groups in 1 gram of solid resin.

Suitable polyesters are derived from a polybasic acid and a polyhydroxy compound and are generally hydroxyl functional.

Polybasic acids are compounds having two or more carboxylic acid groups. Such polybasic acids are well known in the polyester art. Examples of suitable polybasic acids are C₁₋₆ alkane dioic acids such as succinic acid, glutaric acid, adipic acid or hexanedioic acid, cycloaliphatic acids such as hexahydrophthalic acid, unsaturated alkane dioic acids such as fumaric or maleic acids, dimer acids, and aromatic acids such as phthalic acid, isophthalic acid and trimellitic acid. Ester-forming derivatives of such acids can also be used in place of the free acids. Ester-forming derivatives include anhydrides and lower alkyl, for example methyl or ethyl, esters. Mixtures of two or more acids or their ester-forming derivatives can be used.

Polyhydroxy compounds are compounds having two or more hydroxyl groups and are well known in the polyester art. Examples of suitable polyhydroxy compounds are diols such as ethylene glycol, propylene glycol, 1,3-propane diol, butylene glycol, 1,4-butane diol, 1,6-hexane diol, neopentyl glycol, triols such as trimethylol propane, and 5 glycerol, tetrols such as pentaerythritol and higher polyols such as sorbitol. Mixtures of two or more of these polyols can be used.

Polyurethanes or mixed polyester-polyurethanes can be made in a similar way to polyesters but using a di- or polyisocyanate instead of some or all of the polybasic acid. They are generally hydroxyl functional. Suitable diisocyanates are tetramethylxylene 10 diisocyanate, hexamethylene diisocyanate, isophorone diisocyanate and toluene diisocyanate. Suitable polyisocyanates include the isocyanurate trimers, allophanates and uretdiones of diisocyanates such as those described above as well as the reaction products of these diisocyanates with polyols. Polyols are compounds having two or more hydroxyl groups. Suitable polyols include trimethylol propane, glycerol and pentaerythritol. Many 15 such polyisocyanates are commercially available, for example under the Desmodur trade mark from Bayer or the Tolonate trade mark from Rhodia.

The polyester, polyurethane or polyester-polyurethane can be made by conventional means. Generally the components of the polyester are melted together or dissolved in a suitable solvent, such as xylene. The melt or solution is then heated so as to 20 remove the water produced in the reaction between the acid and the hydroxyl groups.

When the components are melted together, the water can conveniently be removed using a fractionating column at temperatures of between 150 and 250°C. When the components are dissolved in a solvent the water can conveniently be removed by azeotropic distillation using a Dean and Stark apparatus at the reflux temperature of the solvent. A combination 25 of these methods can be used, initially melting the components together and removing water using a fractionating column and then adding solvent and removing further water using a Dean & Stark apparatus. If a polyisocyanate is to be included to produce a polyester-polyurethane, then this is generally added after reaction of the other components, and at a lower temperature, such as between 50 and 100°C because the isocyanate groups 30 are much more reactive than acid or ester groups.

Acrylic addition polymers are derived from polymerisable ethylenically unsaturated monomers such as vinyl or acrylic monomers and comprise functional units, hydrophilic units and structural units. Whenever referred to herein, the term acrylic monomer refers to esters of acrylic or methacrylic acid. The term (meth) acrylate refers to both the acrylate and methacrylate equally and the term (meth) acrylic acid refers to acrylic or methacrylic acid equally.

Functional units are derived from vinyl or acrylic monomers which have functional groups on them. For example, glycidyl methacrylate can be used to produce a polymer having epoxy functional groups and aminoethyl methacrylate can be used to produce a polymer having amine functional groups. Polymers having hydroxyl functional groups can be made using hydroxyl functional vinyl or acrylic monomers. An example of a hydroxyl functional vinyl monomer is vinyl alcohol. Examples of hydroxyl functional acrylic monomers are hydroxy ethyl (meth) acrylate, hydroxy butyl (meth) acrylate and hydroxy propyl (meth) acrylate.

Other examples of suitable hydroxyl functional monomers are the reaction products of glycidyl (meth) acrylate with mono-carboxylic acids, such as versatic acid and the reaction product of (meth) acrylic acid with monoepoxy compounds such as Cardura ETM (the glycidyl ester of versatic acid; from Shell).

Hydrophilic units are derived from monomers having hydrophilic groups. Examples of monomers having hydrophilic groups are acid functional monomers, such as acrylic acid and methacrylic acid, and amine functional monomers such as dimethylaminoethyl acrylate.

Structural units are derived from monomers which do not have any functional groups which will react with the crosslinker nor any hydrophilic groups. Examples of monomers from which the structural units can be derived are non-functional vinyl monomers and alkyl esters of (meth) acrylic acid.

Examples of suitable non-functional vinyl monomers are styrene, tertiary butyl styrene and alpha-methyl styrene, preferably alpha-methyl styrene. Examples of suitable alkyl esters of (meth) acrylic acid are C₁₋₁₂ alkyl esters such as methyl (meth) acrylate,

ethyl (meth) acrylate, n-butyl (meth) acrylate, t-butyl (meth) acrylate, n-propyl (meth) acrylate and isobornyl (meth)acrylate.

Molecular weight regulators such as mercapto compounds, for example n-octyl mercaptan, can also be added to control molecular weight.

5 Preferred acrylic addition polymers have a theoretical glass transition temperature (Fox Tg) of -30 to 80°C, more preferably -10 to 50°C.

Many suitable acrylic addition polymers are commercially available. They can also be produced by conventional means. The acrylic addition polymer can be produced by contacting a mixture of the appropriate monomers with a polymerisation initiator at a
10 temperature at which polymerisation occurs. For example the monomer mixture can be slowly fed into a solution of initiator held at the polymerisation temperature or the initiator can be fed into a solvent at the polymerisation temperature simultaneously with the monomer mixture.

Suitable initiators are azo initiators such as azodiisobutyronitrile and 2,2' azobis
15 (2-methylbutyronitrile) or peroxy initiators such as benzoyl peroxide.

The pigment is dispersed in water using a suitable pigment dispersant. A suitable pigment dispersant is Dispex GA40™.

The term pigment includes fillers and extenders as well as conventional pigments.

Pigments are particulate materials which impart colour or opacity to the final paint film.

20 Extenders and fillers are usually inorganic materials which can be used to reduce the cost of a formulation or to modify its properties. In particular, fillers and extenders can be used in primers to improve their ability to hide minor surface imperfections and also to make them easier to rub down with sandpaper to provide a smooth surface for subsequent topcoat application. We have found that the process of the present invention is particularly
25 beneficial in the production of primers because they contain a relatively high level of pigment and so the reduction in voc achieved by this process is particularly marked.

Crosslinkers are compounds which react with at least two functional groups on the polymer so as to crosslink the composition. When the functional groups are carboxylic

acid groups an example of a suitable crosslinker is a carbodiimide. When the functional groups are amine groups, examples of suitable crosslinkers are polyepoxides and polyisocyanates. When the functional groups are epoxy groups, examples of suitable crosslinkers are polyamines. When the functional groups on the polymer are hydroxyl groups, examples of suitable crosslinkers are phenol formaldehydes, melamine formaldehydes and polyisocyanates.

Polyisocyanates are preferred crosslinkers. Polyisocyanates are compounds having two or more isocyanate groups per molecule and are well known in the coatings art. The isocyanate groups can be blocked but unblocked isocyanates are preferred.

10 Suitable diisocyanates are tetramethylxylene diisocyanate, hexamethylene diisocyanate, toluene diisocyanate and isophorone diisocyanate. Preferably, the polyisocyanate has an isocyanate functionality of 2.5 or more isocyanate groups per molecule. Suitable polyisocyanates of this kind include the isocyanurate trimers, allophanates and uretdiones of diisocyanates such as those described above as well as the
15 reaction products of these diisocyanates with polyols. Polyols are compounds having three or more hydroxyl groups. Suitable polyols include trimethylol propane, glycerol and pentaerythritol. Many such polyisocyanates are commercially available, for example under the Desmodur trade mark from Bayer or the Tolonate trade mark from Rhodia.

The aqueous medium comprises at least 50% by weight water, preferably at least 90% and
20 most preferably substantially all, for example 99.5%, water. The other components of the aqueous medium can be water miscible organic solvents.

The compositions can also contain catalysts for the reaction between the functional groups on the polymer and the crosslinker. For example, suitable catalysts for the isocyanate-hydroxyl reaction include tin catalysts such as dibutyl tin dilaurate and amine catalysts
25 such as triethylamine. The compositions can also contain other conventional paint additives such as, pigments, fillers, UV absorbers and flow aids.

The solution of polymer can be made either by making the polymer in organic solvent or by simple stirring of the polymer with the organic solvent.

The pigment millbase can be made by mixing the pigments, fillers and extenders and the pigment dispersant, optionally with some water in a high shear mixer such as a ball mill, or a high speed rotary disperser so as to thoroughly separate the pigment particles and coat them with dispersant.

5 The crosslinker can be dissolved in the organic solvent by mixing, for example by stirring.

The solventborne mixture of polymer, crosslinker and dispersed waterborne millbase can be dispersed in the aqueous medium by stirring.

The coating composition of the invention can be applied to the surface of a substrate and then allowed or caused to dry and cure. According to the present invention there is

10 provided a process for coating a substrate which comprises the steps of applying a layer of a coating composition according to the present invention to a surface of the substrate and thereafter causing or allowing the layer to cure.

The coating composition can be applied by conventional means such as by brush, roller or spray, preferably spray. The substrate can be for example, metal, plastic, wood or glass.

15 The compositions are particularly useful for refinishing motor vehicles when they can be used as topcoats although they are especially useful as primers.

The applied layer can be allowed to cure at ambient temperature in the case where the polymer and crosslinker react together at ambient temperatures. Alternatively the layer can be baked at elevated temperatures, for example 50-120°C to accelerate curing. Drying and
20 curing typically takes between 5 minutes and 24 hours depending on the ambient conditions and on the particular components used. Conveniently it takes about 15 minutes to about 5 hours.

According to the present invention there is also provided a coated article obtainable by the process.

25 The invention will now be illustrated by means of the following examples in which all parts are by weight.

EXAMPLES

1. Waterborne Pigment Millbase.

A dispersant (Dispex GA40™, 2.991 parts), antifoaming agent (Synperionic DF210™, 0.300 parts) and water (11.762 parts) were mixed together and put into a high speed
5 disperser. The disperser was turned on and zinc oxide (2.054 parts), zinc phosphate (14.266 parts), talc (24.414 parts), calcium carbonate (16.270 parts), titanium dioxide (Tipure R960-09™, 21.563 parts) and Organic Black dispersion (Sandosperse™, 0.498 parts) were added steadily so as to avoid the formation of lumps. Water (5.882) was added with the pigment to maintain a fluid mixture. High speed stirring was continued for 45 to
10 60 minutes. Mixing was continued for a further 5 minutes.

Hydroxyl Functional Acrylic Polymer

A mixture of alpha methyl styrene (6.65 parts), tertiary butyl acrylate (19.96 parts), hydroxybutyl acrylate (19.16 parts), butyl acrylate (11.99 parts), acrylic acid (2.14 parts), azidiisobutyronitrile (Vazo 67™, 2.57 parts), n-octyl mercaptan (2.33 parts) and butyl
15 acetate 7.18 parts) were slowly added to a mixture of butyl acetate (21.14 parts and alpha methyl styrene (6.65 parts) over 180 minutes at a temperature of 135°C. The mixture was held at 135°C for 15 minutes and then further initiator (Triganox 21S™, 0.115 parts) was added. The mixture was stirred for a further 60 minutes and then further initiator (Triganox 21S™, 0.115 parts) was added. The mixture was held at 135°C for a further 60 minutes
20 before being allowed to cool.
The hydroxyl functional polymer had an acid value of 28.

3. Pigmented Paint Pack

A mixture of dimethylaminoethanol (0.57 parts) and water (5.16 parts) was added to the polymer solution produced in 2 above (15.77 parts). This mixture was added to the pigment millbase prepared in 1 above (73.87 parts) with stirring followed by butyl acetate
25 (4.57 parts), and dibutyl tin dilaurate catalyst (0.06 parts) .

4. Primer Compositions.

Three primer compositions according to the invention were made with three different polyisocyanate hardeners. The components are set out in Table 1 below;

| Composition | Polyisocyanate Hardener | Pigmented Paint Pack | Hardener | Water |
|-------------|-------------------------|----------------------|----------|-------|
| 1 | Tolonate HDT90 | 50 | 3.05 | 5 |
| 2 | Desmodur Z4470 | 50 | 5.15 | 7.1 |
| 3 | Cythane 3174 | 50 | 5.8 | 7 |

5. Comparative Tests

Two primer compositions were made, Composition 4 according to the invention and comparative composition 5 using hydrophilically modified polyisocyanates which was added to the aqueous composition after mixing of the millbase and water. These compositions used the pigmented paint pack from 3 above (300 parts). For composition 4, a non-hydrophilically modified polyisocyanate, Cythane 3174™ (34.8 parts) was added to the paint pack with stirring and then water (43.8 parts) was added with stirring to give the final paint composition 4. For comparative composition 5, water (43.8 parts) was added to the paint pack and then a hydrophilically modified polyisocyanate Desmodur VPLS 2032™ (39.6 parts) was added with stirring.

These primer compositions were applied by spray to steel panels to give a dry film thickness of 80 microns in three coats. The panels were left for 24 hours at ambient temperature (about 20°C) to dry and cure.

The dried primer coatings were sanded to a smooth finish with P300 paper and a mechanical sander, and then topcoated with a commercial black two-pack polyurethane topcoat. This was allowed to dry at ambient temperature for 1 hour and then stoved for 30 minutes at 60°C.

The primed and topcoated panels were tested for Konig hardness and Watersoak Resistance. Watersoak Resistance is measured according to SMMT (Society of Motor Manufacturers Test) number 57. The results were as follows;

| | Appearance | SMMT Watersoak | Adhesion |
|----------------------|----------------------|----------------|----------|
| Primer 4 | Smooth & Glossy | 8M | 33 |
| Comparative Primer 5 | Glossy with Pinholes | 8VD | 1 |

The Watersoak Resistance test result indicates the blistering of the paint after 3 days. M 5 means Medium blisters and VD means Very Dense blisters. The blister size of 8 is on a scale of 1 to 10 where 1 is 10mm and 10 is microscopic. Adhesion indicates the percentage of coating remaining intact after applying sticky tape to the tested topcoat and removing it.

This Watersoak Resistance test very clearly shows the benefit of the process and composition according to the invention which avoids the need for hydrophilically modified polyisocyanates while giving a much lower voc than is achievable by using known solventborne millbases in conjunction with non-hydrophilically modified polyisocyanates.

6. Topcoat Composition.

A white topcoat according to the invention was prepared as follows

15 White Waterborne Pigment Millbase.

A dispersant (Dispex GA40™, 26.5 parts), antifoaming agent (Synperionic DF210™, 9 parts), polypropylene glycol (100.0 parts) and water (150.0 parts) were mixed together and put into a high speed disperser. The disperser was turned on and titanium dioxide (Tipure R960-09™, 1393.0 parts) was added steadily so as to avoid the formation of lumps. Water 20 (183.0 parts) was added with the pigment to maintain a fluid mixture. High speed stirring was continued for 45 to 60 minutes. Mixing was continued for a further 5 minutes.

White Pigmented Paint Pack

A 10% aqueous solution of dimethylaminoethanol (7 parts) was added to the polymer solution produced in 2 above (200.00 parts). This mixture was added to the white pigment prepared above (190.0 parts) with stirring followed by butyl acetate (20 parts), and dibutyl tin dilaurate catalyst (0.25 parts).

5 White Topcoat Composition.

A white topcoat was made by mixing the paint pack prepared as above (164 parts), a polyisocyanate (Cythane 3174™, 46.0 parts), a 10% aqueous solution of dimethylaminoethanol (14.0 parts) and water (200.0 parts).

Application and testing

- 10 The paint was allowed to stand for 30 minutes after mixing to give a low viscosity composition suitable for spraying. The composition was sprayed onto a primed steel panel at a 40 micrometer dry film thickness. It was left to dry for 1 hour at ambient temperature (about 20°C) and then stoved for 30 minutes at 60°C.

The resulting topcoat was smooth, glossy and defect free with a gloss of 70 at 20 degree
15 angle and a Koenig hardness of 53.

CLAIMS

1. A pigmented solventborne paint pack which can be made into a waterborne coating composition which comprises;
 - i) a waterborne pigment dispersion comprising pigment dispersed in water in the presence of a pigment dispersant, the aqueous pigment dispersion itself being in dispersion in
 - ii) a solution in an organic solvent of polymer having functional groups and hydrophilic groups.
2. A paint pack as claimed in claim 1 in which the functional groups are hydroxyl groups.
3. A paint pack as claimed in claim 2 in which the polymer has a hydroxyl value of 5 to 500
4. A paint pack as claimed in claim 3 in which the polymer has a hydroxyl value of 50 to 250.
5. A paint pack as claimed in any preceding claim in which the hydrophilic groups are carboxylic acid groups or amine groups.
6. A paint pack as claimed in claim 5 in which the hydrophilic groups are carboxylic acid groups and the polymer has an acid value of 20 to 250.
7. A paint pack as claimed in claim 5 in which the hydrophilic groups are amine groups and the polymer has an amine value of 20 to 250.
8. A paint pack as claimed in any preceding claim in which the polymer is a vinyl addition polymer, a polyester, a polyurethane, a mixed polyester-polyurethane or an epoxy polymer.
9. A paint pack as claimed in claim 8 in which the polymer is a vinyl addition polymer, a polyester, a polyurethane or a mixed polyester-polyurethane.

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10. A paint pack as claimed in claim 9 in which the polymer is a vinyl addition polymer.

11. A paint pack as claimed in claim 10 in which the polymer has a theoretical glass transition temperature (Fox T_g) of -30 to 80°C.

12. A paint pack as claimed in claim 11 in which the polymer has a theoretical glass transition temperature (Fox T_g) of -10 to 50°C.

13. A paint pack as claimed in any preceding claim in which the polymer has a number average molecular weight as measured by gel permeation chromatography of 700 to 10,000.

14. A paint pack as claimed in claim 13 in which the polymer has a number average molecular weight of 1,000 to 4,000.

15. A paint pack as claimed in any one of the preceding claims in which the polymer has an acid value of up to 50.

16. A solventborne activated paint pack which comprises the solventborne paint pack as claimed in any one of claims 1 to 14 and a crosslinker which is dissolved in the organic solvent.

17. A solventborne activated paint pack as claimed in claim 16 in which the crosslinker is a phenol formaldehyde, melamine formaldehyde, or polyisocyanate.

18. A solventborne activated paint pack as claimed in claim 17 in which the crosslinker is a polyisocyanate.

19. A waterborne coating composition which comprises a dispersion in an aqueous medium of the solventborne activated paint pack as claimed in claim 16.

20. A process for producing a solventborne paint pack which can be made into a pigmented waterborne coating composition, comprising a polymer having hydrophilic groups and functional groups, and a crosslinker for the polymer, comprising the steps of;

- i) forming a solution of the polymer in an organic solvent
- ii) dispersing a waterborne pigment dispersion in the polymer solution.

21. A process for producing a solventborne activated paint pack which can be made into an aqueous coating composition comprising the process of claim 20

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and the further step of adding to the solventborne paint pack a crosslinker which is soluble in the organic solvent and forming a solution of the crosslinker in the solvent.

22. A process for producing an aqueous coating composition which comprises the process of Claim 21 and the further step of emulsifying the solventborne activated paint pack in an aqueous medium.

23. A process for coating a substrate which comprises the steps of applying a layer of a waterborne coating composition according to claim 22 to a surface of the substrate and thereafter causing or allowing the layer to cure.

24. A coated article obtainable by the process of claim 23.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/04375

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C09D17/00 C08G18/62

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C09D C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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| X | WO 96 30425 A (PPG INDUSTRIES) 3 October 1996 (1996-10-03) page 2, line 28 -page 7, line 30; claims 1,7,17; examples | 1,5, 16-24 |
| X | DE 42 26 270 A (HERBERTS) 10 February 1994 (1994-02-10) page 2, line 32 -page 9, line 65; claims 1-11; examples | 1-6, 8-10,13, 16-24 |
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

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"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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